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AN ADHESIVE FOR FIELD REPAIR OF COMPOSITES

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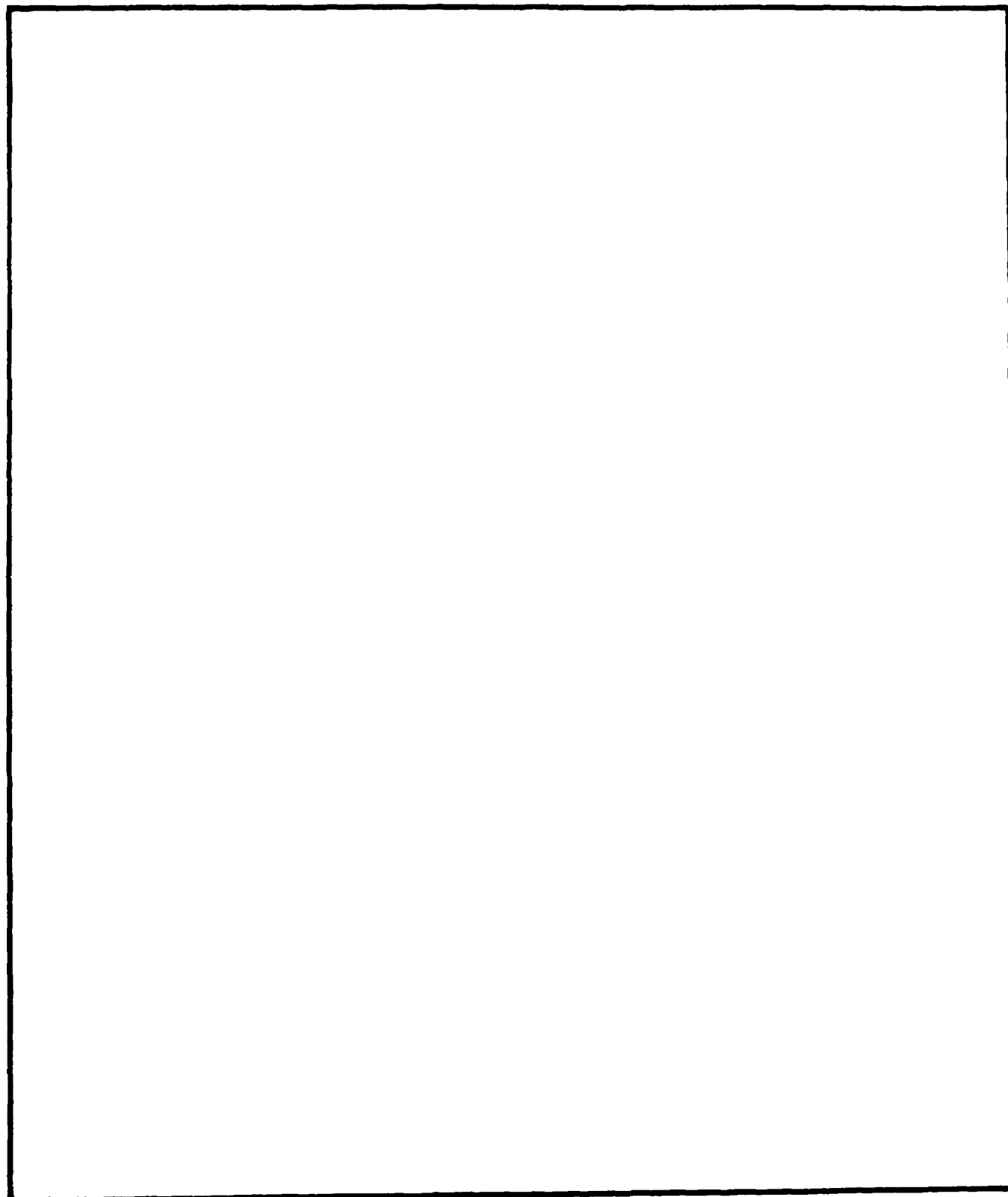
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BACKGROUND

The increased use of composites on Naval aircraft and the need to repair these components at field locations has created a requirement for an ambient storage adhesive. Drawbacks of currently available materials include a need for refrigerated storage, short shelf lives even at 0 F and failure to meet mechanical and thermal property requirements (1). The primary goal of this program is to develop a two-part liquid adhesive which can easily be used in a field repair situation. The requirements of the adhesive would typically be: ambient storage for at least 1 year, short time low temperature cure, vacuum bag pressure consolidation, service use between -65°F and 220°F, and mechanical properties similar to state of the art adhesives.

An adhesive meeting most of these requirements was formulated under contract by Northrop Corp (2). The adhesive consisted of a diglycidal ether of bisphenol A (DGEBA) type epoxy, Epon 828, toughened with carboxy terminated butadiene nitrile rubber, (CTBN), and cured with a cycloaliphatic diamine, diamino dicyclohexyl methane (PACM-20). The adhesive possessed ambient storage capability, environmental durability and the required mechanical properties. A cure cycle of 1 hour at 300 F was used to provide thermal stability in the system.

This adhesive was subsequently used in full scale repair demonstrations. In these tests the adhesive was used to bond a composite patch to a composite laminate. The repair was performed with vacuum bag pressure and a heating blanket. Testing of these repair specimens showed that the bondlines had a high void content. The large number and size of the voids limited the structural integrity of the repair. A process modeling study was initiated to investigate the cause of this voiding.

The processing study involved the development of a theoretical model of adhesive flow during cure (3). This study showed that when sufficient moisture is present in the adhesive, either from the adhesive or from diffusion from wet substrates during cure, the factor controlling void formation is the hydrostatic pressure of the fluid in the bondline. Figure 1 illustrates the case studied. Here an external pressure, P_a , is applied to a patch while the edge of the patch is exposed to a pressure, P_o . If an external load is used the external pressure, P_a , will be atmospheric pressure, plus the mechanical load divided by the patch area and the edge pressure P_o , will be atmospheric pressure. For a repair carried out in a vacuum bag, the external pressure will be atmospheric, while the edge pressure will be the vacuum bag pressure (close to zero). In order to achieve equilibrium, adhesive will flow from the center of the patch toward the edge. This produces a hydrostatic pressure gradient in the adhesive. It was shown that for a long thin rectangular patch, adhesive flowed from a peak pressure at the center of $3/2 (P_a - P_o)$ to the edge. The pressure P_x at a distance x from the center line is given by Equation 1.

$$P_x - P_o = \frac{6\eta}{Z^2} \frac{dz}{dt} \left[\frac{X_o^2}{4} - x^2 \right] \quad (1)$$

Where Z is the bondline thickness at time t , when η is the viscosity of the adhesive and X_o is the width of the patch. This pressure distribution does not change with time until the bondline thickness becomes comparable with the surface roughness. At this point local hydrostatic pressure gradients exist between adhesive sites adjacent to high and low spots on the roughened surface. In the limit, the patch comes to rest on these high spots. The pressure on the patch is distributed through the points of contact leaving the surrounding area at low pressure. In the low pressure areas volatilization of water becomes possible as the temperature is increased.

In order to prevent the volatilization of water during the cure, the hydrostatic pressure in the fluid must be maintained as high as possible until gelation occurs. This means that the patch must be prevented from settling onto the substrate surface or from being supported by scrim or fillers in the adhesive. An ideal adhesive according to the model would have a low gel temperature (below 100°C), and a relatively high viscosity, in excess of 100 poise throughout the cure cycle. Rheometrics data for the adhesive formulated at Northrop showed that the material gelled at approximately 90°C with a minimum viscosity of 4 poise (Fig. 2).

At this point an effort was initiated to increase the minimum viscosity of the adhesive from 4 to 100 poise. Several methods of increasing the viscosity were evaluated including holding the adhesive at a low pressure until the viscosity increased through reaction, adding a filler to increase viscosity, and adducting each or both parts of the adhesive. Adducting is a process in which a small amount of each component is pre-reacted with the other component at elevated temperature. The result is a higher viscosity adhesive with mechanical properties similar to the original adhesive. The viscosity of the adducted components can be controlled by the ratio of resin and curing agent used to make the adduct. Adducting proved to be the best method for increasing the viscosity since it did not effect mechanical properties and did not require the operator to continuously monitor the cure.

EXPERIMENTAL

The first adducted adhesives were made by mixing 80 grams of EPON 828, 18 grams CTBN and between .28 and 3.4 grams of PACM-20. The mixture was heated to 160°C at which time .2 grams of triphenyl phosphene was added. The mixture was then heated to 170°C and held for 1 hour. The curing agent adducts were made by mixing 100 grams of PACM-20 with between 1 and 12 grams of the 828-CTBN mixture. This adduct was allowed to react overnight at room temperature. A second set of adducted adhesives was made by mixing 80 grams of DER-332 with 18 grams of CTBN heating to 160°C and adding .2 grams of triphenyl phosphene. The mixture was heated to 170°C and held for 1 hour. After the hold 1.67 grams of Amicure PACM were added and the mixture held at 170°C for 1 hour. The curing agent or B side adducts were made by mixing 23, 27 and 32 grams of 332 with 100 grams of Amicure PACM. The hardener adducts were heated at 120°C for 1 hour to complete the reaction. The viscosities of these adducts was measured using a Brookfield Viscometer.

After evaluation of the data from the first 2 adducted adhesive sets a third adduct was made. The A side of this adduct contained 80 grams of 332 and 18 grams of CTBN. This mixture was heated to 160°C, .2 grams of triphenyl phosphene were added and heating continued to 170°C. After 1 hour hold at 170°C, 1.67 grams of Amicure PACM were added. The B side adduct was made by mixing 340 grams of Amicure PACM with 100 grams of 332. The mixture was heated to 120°C and held for 1 hour.

The physical and mechanical properties of this adduct were fully characterized. Brookfield model RVTDV-11 and a Rheometrics model RD57700 viscometers were used to determine the viscosities of the unmixed and mixed adhesive. Rheometrics were run at 2°C/min heatup rate to simulate a cure cycle. Castings of the adhesive were made to determine the glass transition temperature of dry and saturated adhesive. Samples were run at 10°C/min on a DuPont 982 Dynamic Mechanical Analyzer. A Du Pont 911 Differential Scanning Calorimeter was used to measure the size of the cure exotherm and the temperature of the peak of reaction.

The mechanical properties of the adducted adhesives were determined from single, double, and thick adherend lap shear testing (ASTM-D-1000, ASTM-D-3983). Floating roller peel (ASTM 3167) and climbing drum peel (ASTM D1781) testing were also performed. All testing was performed at room temperature, 104°C and 104°C/wet. Wet samples were exposed for 30 days in a humidity chamber at 95% RH and 60°C. Static and dynamic durability testing (ASTM D2912) was also performed. In this test, specimens were loaded to 1500 PSI in a humidity chamber set at 95% RH and 60°C. The dynamic

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specimens were loaded for 50 minutes and unloaded for 10 minutes. Static specimens were exposed for 90 days; the dynamic samples for 2100 hours.

In addition to coupon testing, full scale repairs were conducted. An 8-ply woven quasisisotropic patch was bonded over a 2 inch hole in a 50/40/10 24-ply graphite/epoxy laminate. Seven specimens were prepared. Six of the 24 ply laminates were saturated to a 1% moisture level before the patches were bonded. Two specimens were tested at room temperature, two at 104°C dry and two at 104°C after 30 days in humidity chamber. The seventh laminate was repaired without moisturizing and tested at room temperature. After laboratory demonstration of the adhesive performance, the adhesive was provided to NARF, Cherry Point MCAS for field evaluation.

RESULTS AND DISCUSSION

Since there was no information on the level of adducting required to produce an adhesive with a minimum viscosity of 100 poise, the first set of adducted adhesives were formulated to evaluate the range of viscosities possible. The physical and mechanical properties were measured to determine the effect of adducting on adhesive response. This set of adducts was made by mixing equal weight percentages (1-12%) of resin and curing agent with the respective components. Viscosity of the resin and curing agent as well as the minimum viscosity as measured by Brookfield viscometer is given in TABLE 1 for the 1.7 (6%) to 2.55 (9%) adducts (of both adhesive parts). Adduct formulations below 6% had minimum viscosities well below 100 poise and were eliminated from further evaluation. Adducts above 9% had such high viscosities that they were difficult to mix. These adducts were eliminated at this time. Adhesive formulations between 6 and 9% were used to bond lap shear and peel specimens. Testing was performed at room temperature and 104°C. Results of coupon testing showed that there was no degradation of mechanical properties of the adhesive due to adducting (TABLE 2). The adhesive adducts in the 6 to 9% range met the viscosity and mechanical property requirements of the program. One problem that was encountered during evaluation was that the order of magnitude of difference in viscosity between adducted resin and curing agent components made the adhesive difficult to mix. At this time a second set of adducts were made in an effort to increase the viscosity of the curing agent to the same level as the resin. These curing agents adducts incorporated much more of the resin than had been done previously. The levels examined were 23, 27 and 32 percent respectively. This information was used to determine the adduct ratio needed to produce a curing agent with the same viscosity as that of the resin. The data were plotted as shown in Figure 3, and the composition required was determined by interpolation.

The third adducted adhesive produced contained 1.67 grams of PACM and had a viscosity of 1500 poise. For a resin with a viscosity of 1500 poise, the required adduct ratio for the B side adduct was 29.5 grams resin in the 100 grams of curing agent. Rheometrics viscosity determinations were made on mixed adhesive samples heated at 2°C/minute. A typical viscosity profile of the adducted adhesive (Fig. 4) shows a mixed viscosity of 1000 poise dropping to a minimum of 98 poise at 70°C.

Several batches of adducted adhesive were made containing 80 grams of DER 332, 18 grams of CTBN and .2 grams of triphenyl phosphene. The resin and rubber are mixed and heated to 160°C at which time the triphenyl phosphene catalyst was added. The mixture was heated to 170°C and held for 1 hour. After 1 hour at 170°C, 1.67 grams of amicure PACM are added and reacted for 1 additional hour. The B side adduct is made by mixing 100 grams of Amicure PACM with 29.5 grams of DER 332. The mixture is heated to 120°C and reacted for 1 hour. The A and B sides are mixed in the ratio of 100 parts A to 38.4 parts B. All physical and mechanical testing of the adhesive were performed with the formula and mix ratio.

Differential Scanning Calorimetry (DSC) was used to determine the size of the cure exotherm and the peak temperature of the reaction. A typical DSC plot is given in Figure 5. The cure exotherm is 178

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J gm with a peak at 82.5°C. Dynamic Mechanical Analysis (DMA) of a dry and wet adhesive sample are shown in Figures 6 and 7. The adhesive has a dry glass transition temperature (T_g) of 160°C and a wet T_g of 148°C.

The results of coupon testing are summarized in Table 3. Single, double and thick adherend lap shear as well as peel and durability testing were performed. Single lap shear data for three batches of material are shown. The thick adherend lap shear strength, strain, and modulus are comparable to state of the art film adhesives. The toughness as evidenced by the peel resistance is high at all test temperatures. The static and dynamic durability testing exceeded the requirements. This adhesive met all of the requirements of MIL-A-85705 (Adhesive for Aircraft Repair).

The results of the full scale testing are given in Table 4. In all cases the repairs restored at least 5300 μ in/in strain to the laminate. All of the repairs provided a wide margin of safety over the required 4000 μ in/in strain. Microscopic examination of the bondlines showed no evidence of voiding. In addition to the physical and mechanical property testing the adhesive has been used in several composite repair demonstrations.

In addition to the laboratory testing performed at NADC, field evaluations of the adhesive were performed at Cherry Point. Repairs were made to graphite epoxy parts at Cherry Point MCAS by Marine Corps personnel as part of the Composite Repair Training Course. These repairs were ultrasonically inspected and sectioned to evaluate bondline quality. No evidence of voiding was found.

The adhesive was also used in the AV-8B Horizontal Stabilizer Life Assurance Program (4). In this program a precured and a B staged patch were bonded to a damaged and moisturized AV-8B stabilizer (Figures 8 and 9). The stabilizer was then fatigued for 6 life times. As a final test the stabilizer was subject to critical reversed loading until failure. Failure occurred at 270% of design limit load. The failure did not initiate at either of the bonded repairs.

SUMMARY

The strength, durability, storability, processability and field adaptability of the NADC adhesive have been thoroughly demonstrated, and its stress-strain behavior has been well characterized. This adhesive contains 80 grams of DER 332, 18 grams CTBN and .2 grams triphenyl phosphene. This mixture is reacted at 170°C for 1 hour. After reaction 1.67 grams of Amicure PACM were added and reacted for 1 hour. The B side adduct was made by reacting 29.5 grams of DER 332 with 100 grams of Amicure PACM. Representative repairs have been successfully fabricated using the adducted adhesive system. Under structural evaluation the adhesive provided comfortable margins of safety for field process variability. Field evaluations have demonstrated the safe application of bonded patches under field conditions using existing ground support equipment and maintenance personnel.

The adhesive has been qualified to military specification MIL-A-85705 Adhesive, Aircraft for Structural Repair and has been approved for use on the AV-8B aircraft as a structural repair adhesive.

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- (3) Donnellan, T.M., Williams, J.G., Trabocco, R.E. An Investigation of Void Formation in Adhesive Bonds Produced Under Field Repair Conditions Report No. NADC-84079-60, Feb 84
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TABLE 1
VISCOSITY OF ADDUCT SETS 1 AND 2

SET 1

| <u>Adduct Weight %</u> | <u>Viscosity (poise) (A Side)</u> | <u>Viscosity (poise) (B Side)</u> |
|----------------------------|---------------------------------------|---------------------------------------|
| 6 | 4700 | 130 |
| 7 | 6450 | 420 |
| 8 | 7900 | 1200 |
| 9 | 11200 | 2900 |

SET 2

| <u>Adduct Weight %</u> | <u>Viscosity (poise) (B Side)</u> |
|----------------------------|---------------------------------------|
| 23 | 180 |
| 27 | 530 |
| 32 | 3300 |

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TABLE 2

MECHANICAL PROPERTIES OF ADDUCT SET 1

Single Lap Shear

| <u>Adduct Weight %</u> | <u>Room Temperature</u> | <u>104°C</u> |
|----------------------------|-----------------------------|--------------|
| 0 | 4900 psi | 2870 psi |
| 1.7 | 4750 psi | 3800 psi |
| 2.0 | 4600 psi | 3640 psi |
| 2.25 | 4400 psi | — |
| 2.5 | 4610 psi | 3580 psi |

Peel

| <u>Adduct Weight %</u> | <u>Room Temperature</u> |
|----------------------------|-----------------------------|
| 0 | 24 piw |
| 1.7 | 34 piw |
| 2.0 | 32 piw |
| 2.25 | 35 piw |
| 2.5 | 34 piw |

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TABLE 3

MECHANICAL PROPERTIES OF ADDUCTED NADC ADHESIVE

| LAP SHEAR STRENGTH | | | | BELL PEEL | | |
|-----------------------|-----------------------|-----------------------|------------------------|-----------------------|-----------------------|-----------------------|
| ROOM TEMP (psi) | 104°C DRY (psi) | 104°C WET (psi) | - 55°C DRY (piw) | ROOM TEMP (piw) | 104°C DRY (piw) | 104°C WET (piw) |
| Batch 1 4770 | 3585 | 2522 | 46.8 | 35.7 | 30.6 | 28.1 |
| Batch 2 5140 | 3620 | 3440 | | | | |
| Batch 4 5470 | 3860 | 2600 | | | | |

| THICK ADHEREND LAP SHEAR | | | | DURABILITY | |
|--------------------------|--------------|--------------|--|------------|--|
| ROOM TEMP | 104°C DRY | 104°C WET | | STATIC: | 1500 psi, 140°F, 100% R.H. 2100 hours with no failure |
| STRENGTH (psi) | (psi) | (psi) | | DYNAMIC: | 1500 ps, 140°F, 100% R.H. 300 cycles (one cycle 50 mins on, 10 mins off) with no failures |
| 5023 | 3980 | 3430 | | | |
| STRAIN 0.47 | 1.28 | 0.92 | | | |
| MODULUS (psi) | (psi) | (psi) | | | |
| 91000 | 44300 | 35500 | | | |

| DOUBLE LAP SHEAR | | |
|-----------------------|----------------|-----------------------|
| ROOM TEMP (psi) | 104°C (psi) | 104°C WET (psi) |
| 5500 | 3100 | 2300 |

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Table 4

FULL SCALE TEST RESULTS FOR NADC ADHESIVE

Repair of a 2 inch hole in a 24 ply (50 40 10) laminate using an 8 ply quasiisotropic DMHDA patch.

Repair of a dry laminate tested at room temperature
far field strain 6700 micro in in

Repair of a moisturized laminate tested at room temperature
far field strain 5540 micro in in

Repair of a moisturized laminate tested at room temperature
far field strain 7000 micro in in

Repair of a moisturized laminate tested at 220°F
far field strain 5320 micro in in

Repair of a moisturized laminate tested at 220°F
far field strain 5880 micro in in

Repair of a moisturized laminate tested at 220°F after exposure to 48 days at 160 F and 95% RH
far field strain 6570 micro in in

Repair of a moisturized laminate tested at 220°F after exposure to 63 days at 160 F and 95% RH
far field strain 6580 micro in in

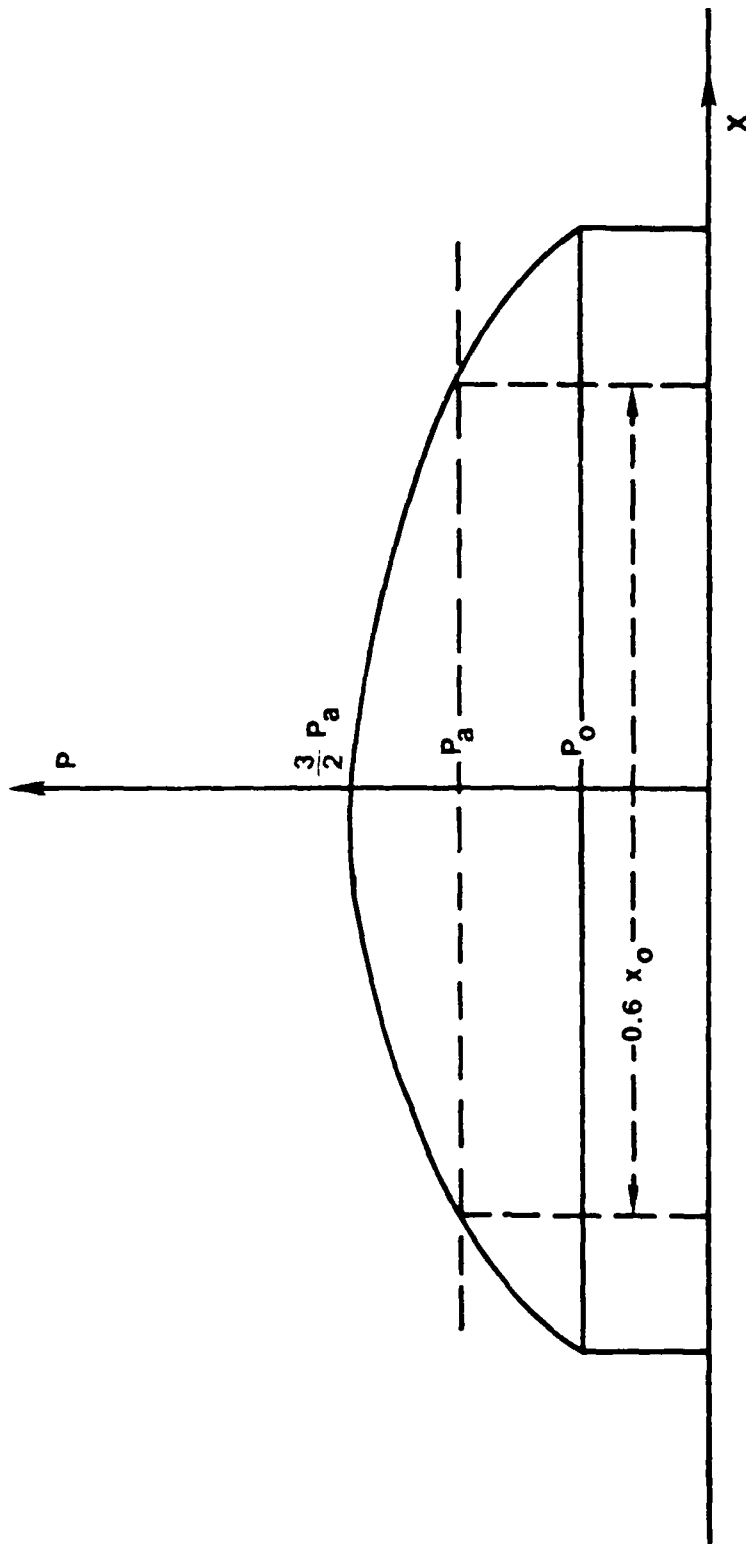


Figure 1. Predicted Pressure Distribution Under Patch

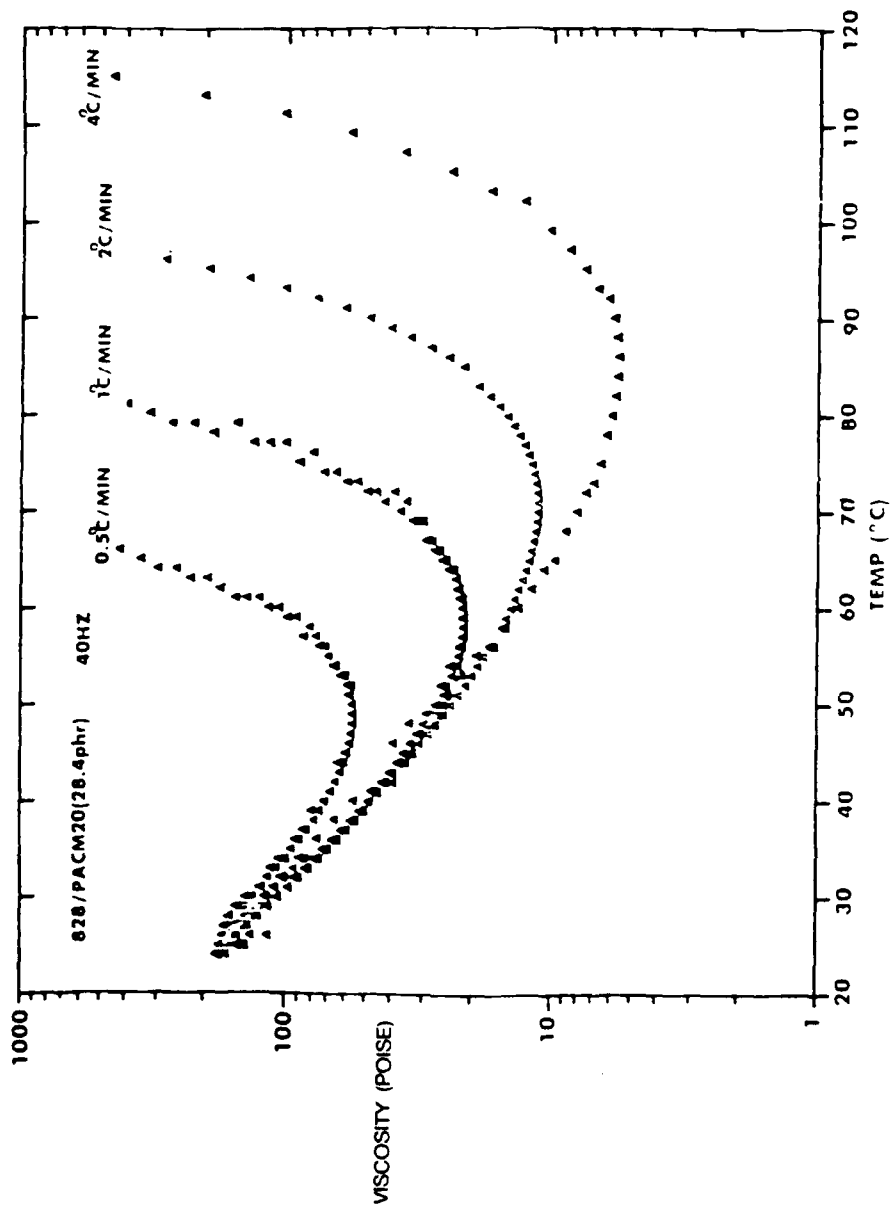


Figure 2. Viscosity of NADC Adhesive

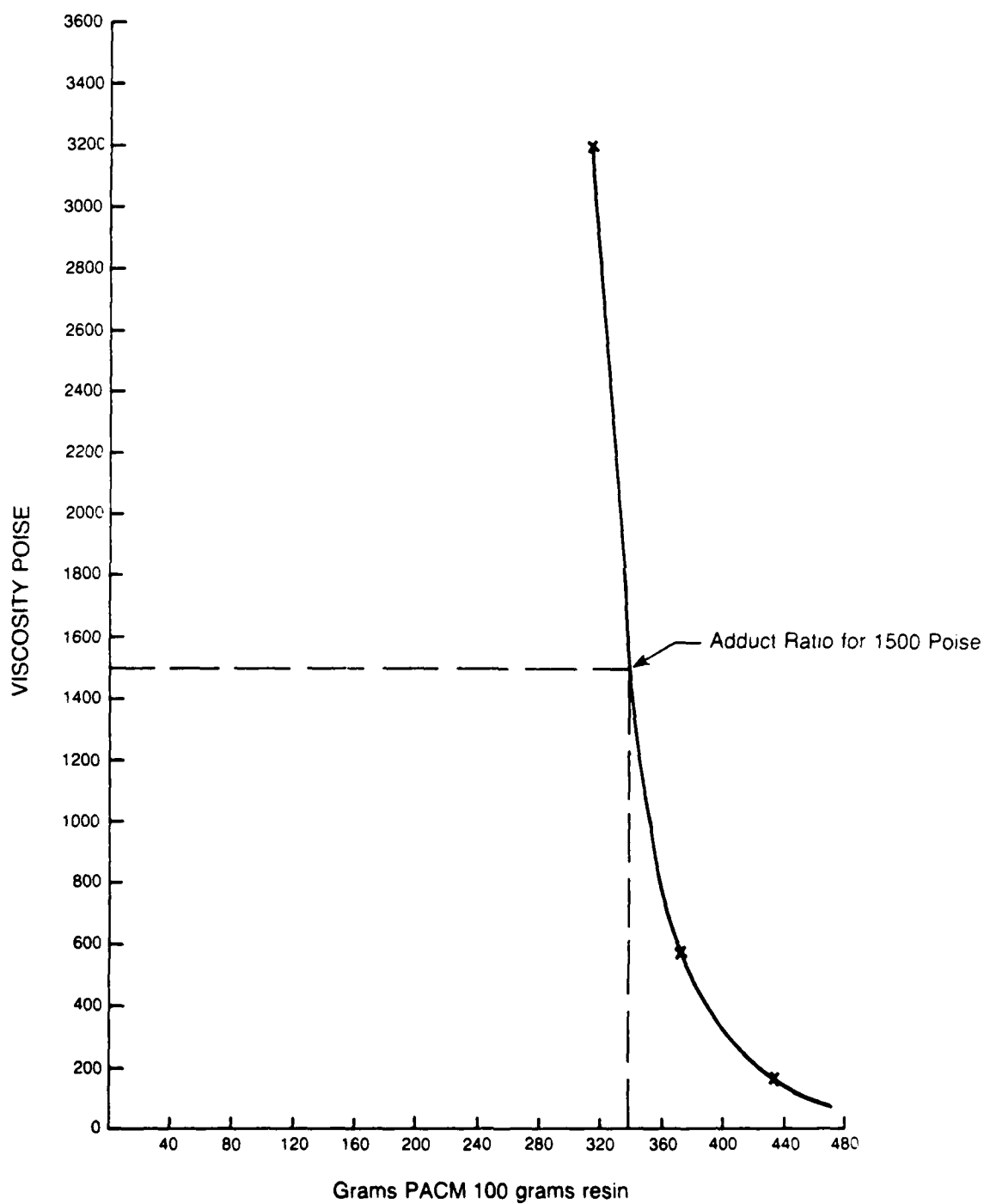


Figure 3. B Side Viscosity

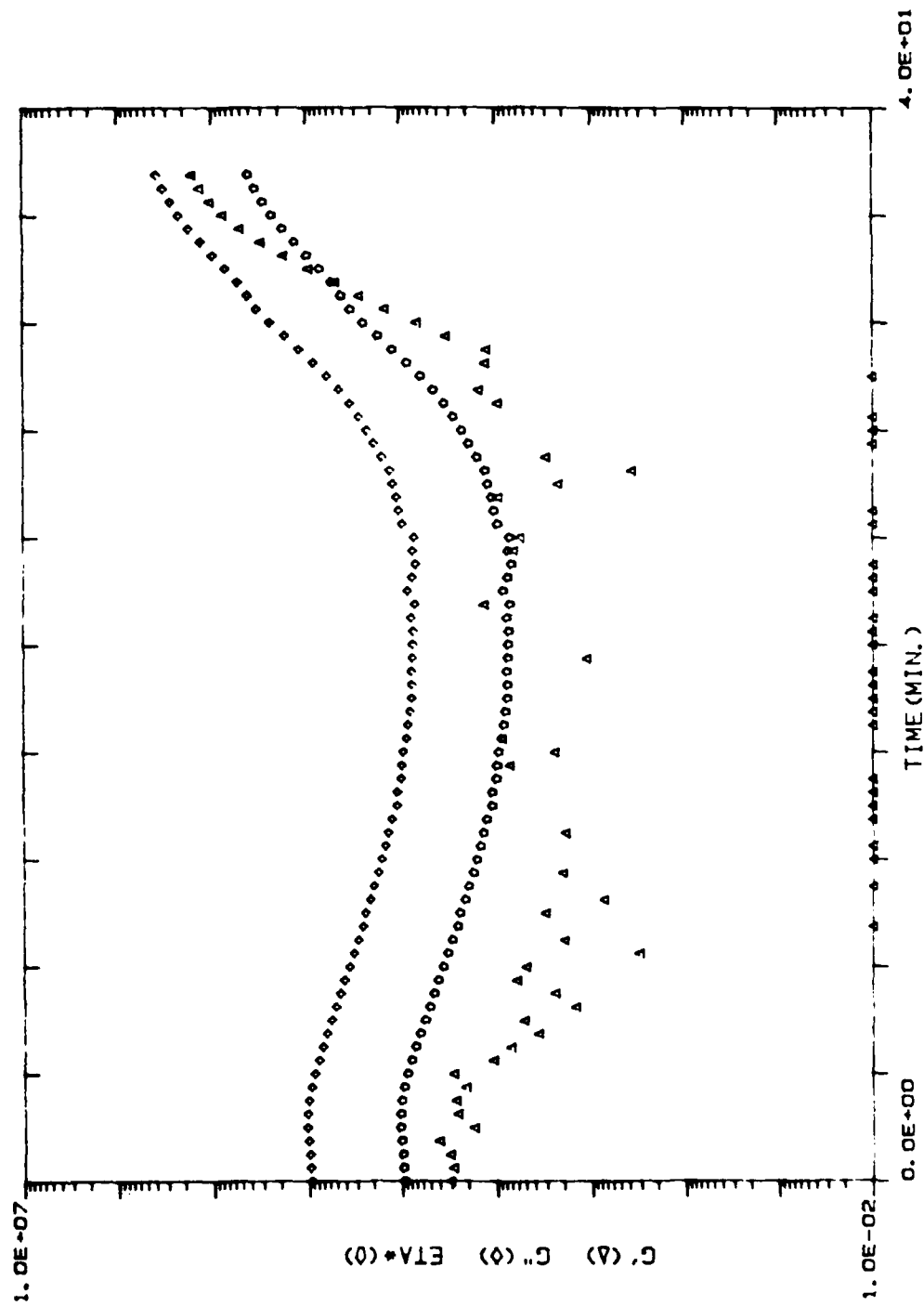


Figure 4. Viscosity of NADC Adhesive Heated at 2°C/min.

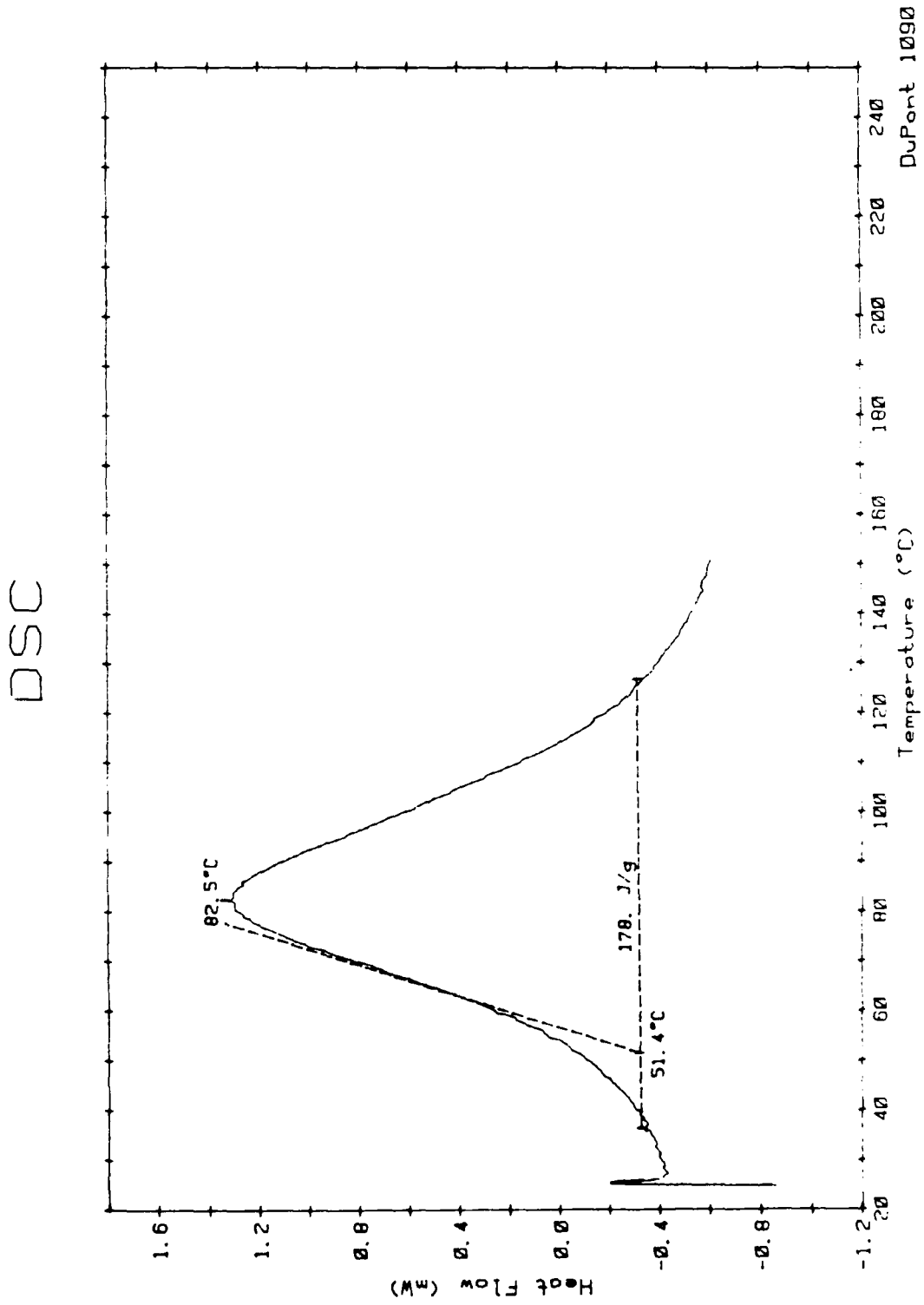


Figure 5. DSC plot of NADC Adhesive 5°C/min.

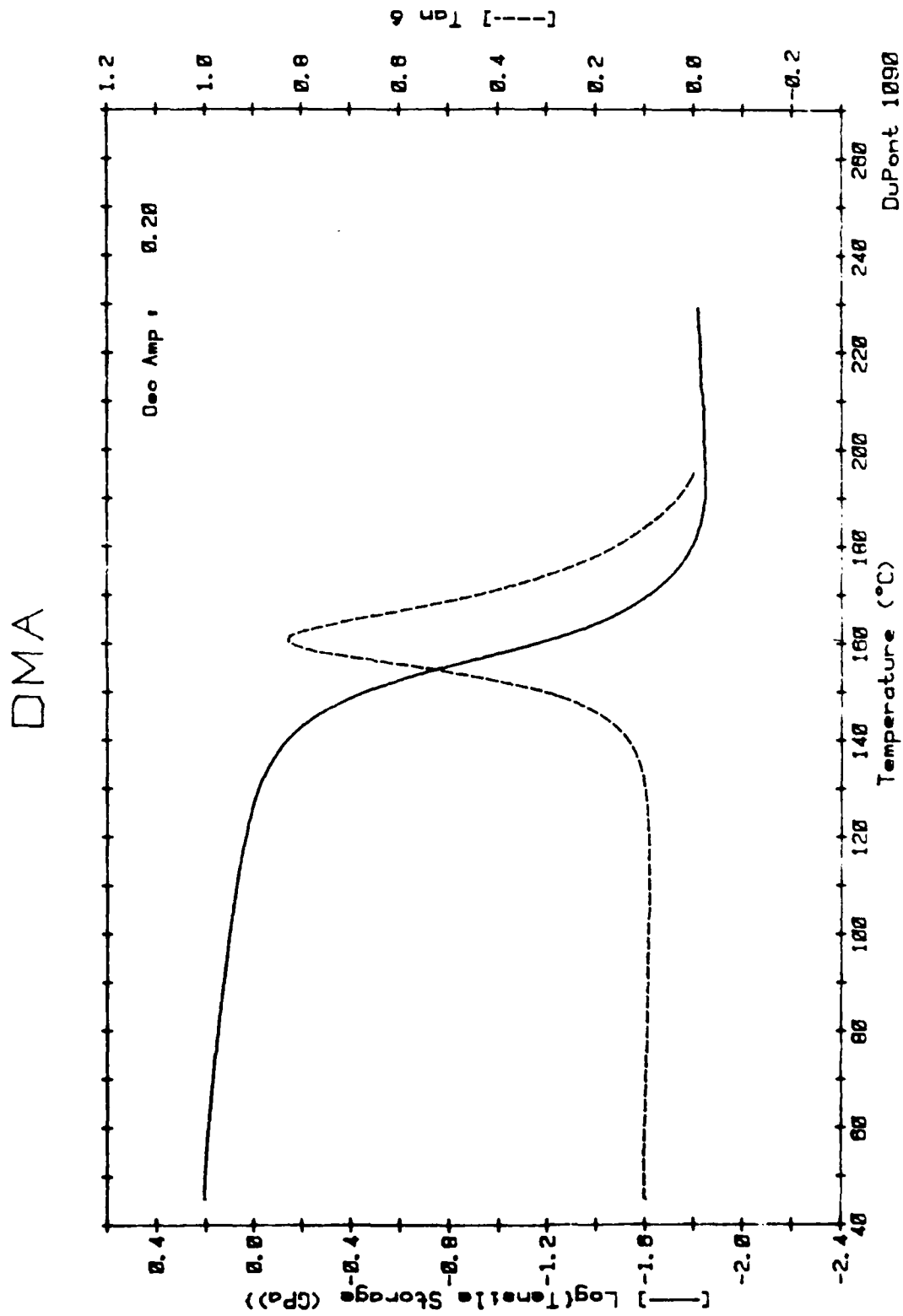


Figure 6. DMA plot for Dry Adhesive

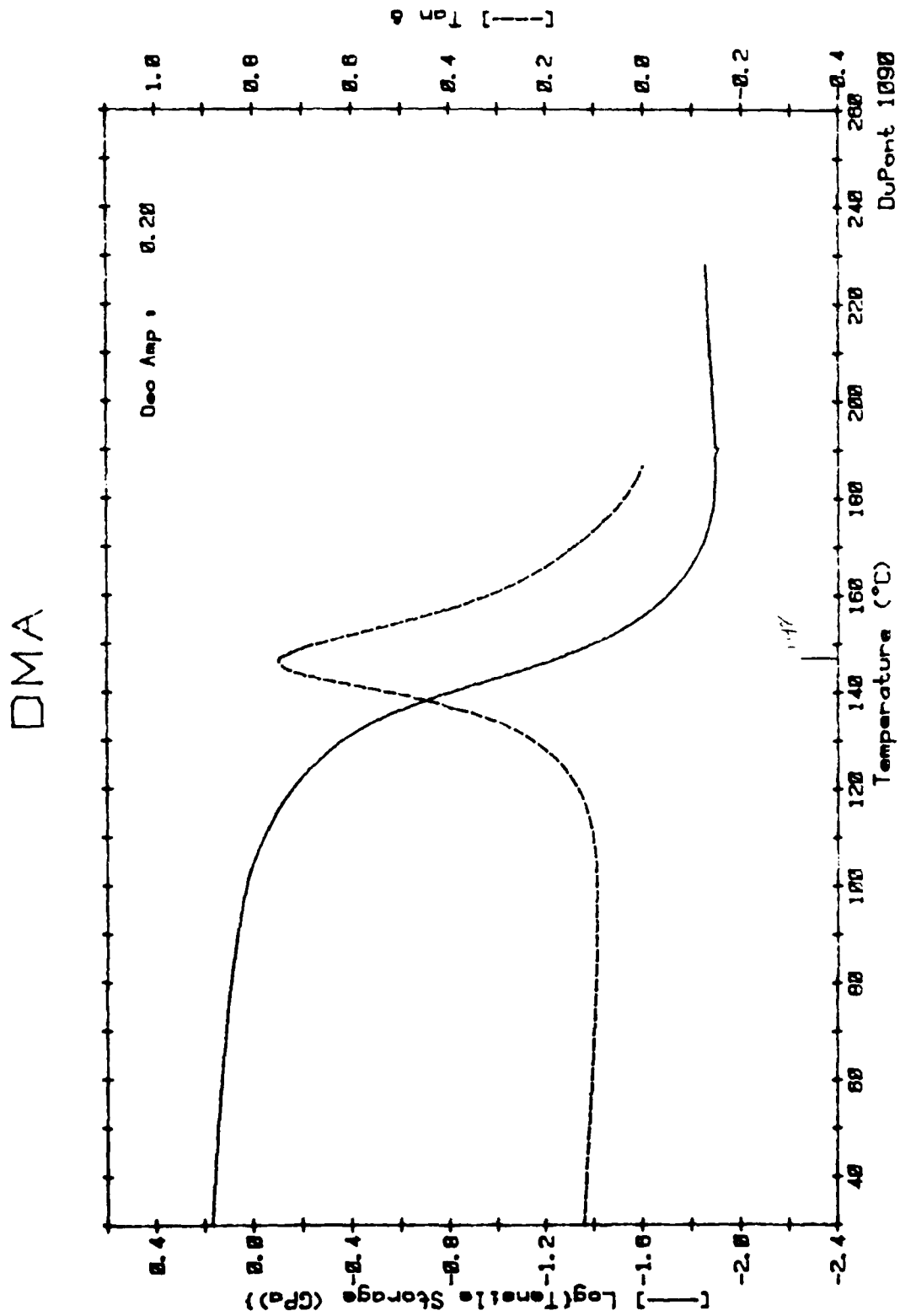


Figure 7. DMA plot for Wet NADC Adhesive

Trailing Edge Repair

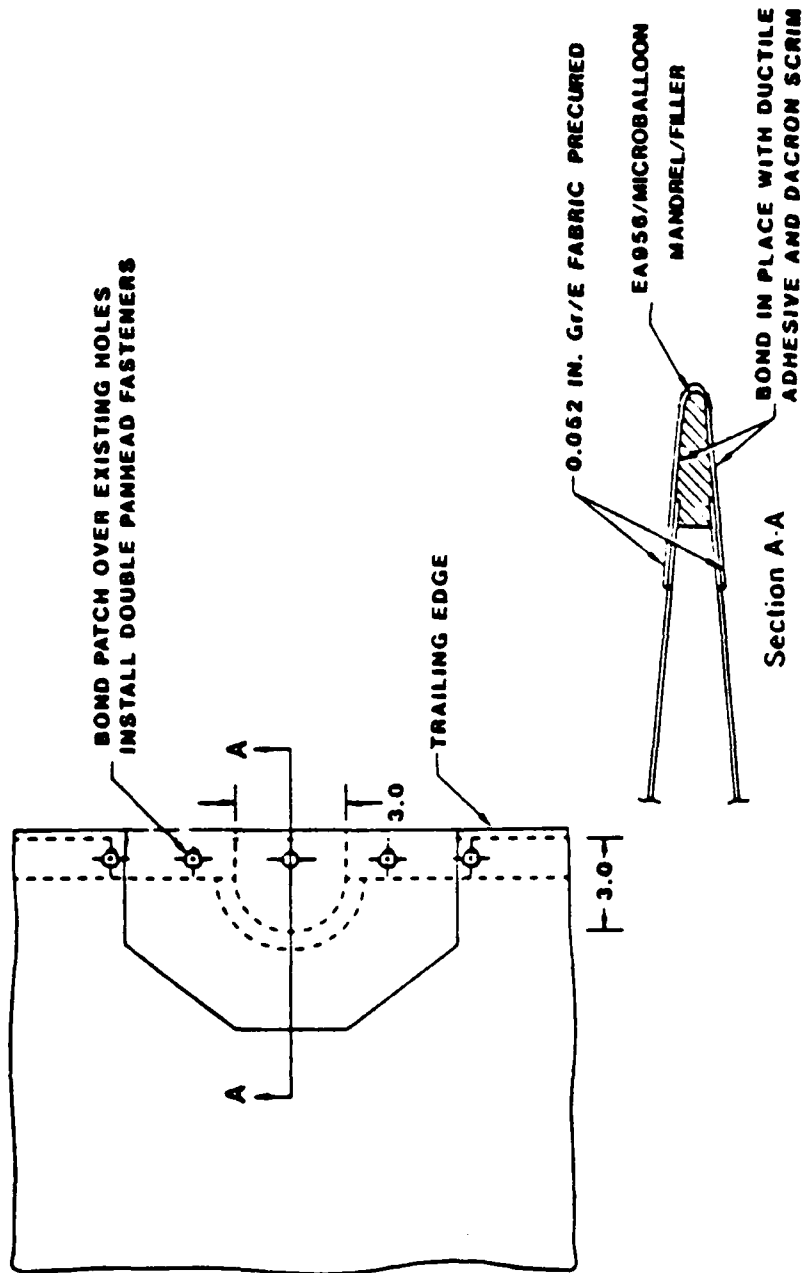


Figure 8. Precured Trailing Edge Repair

External Bonded Repair

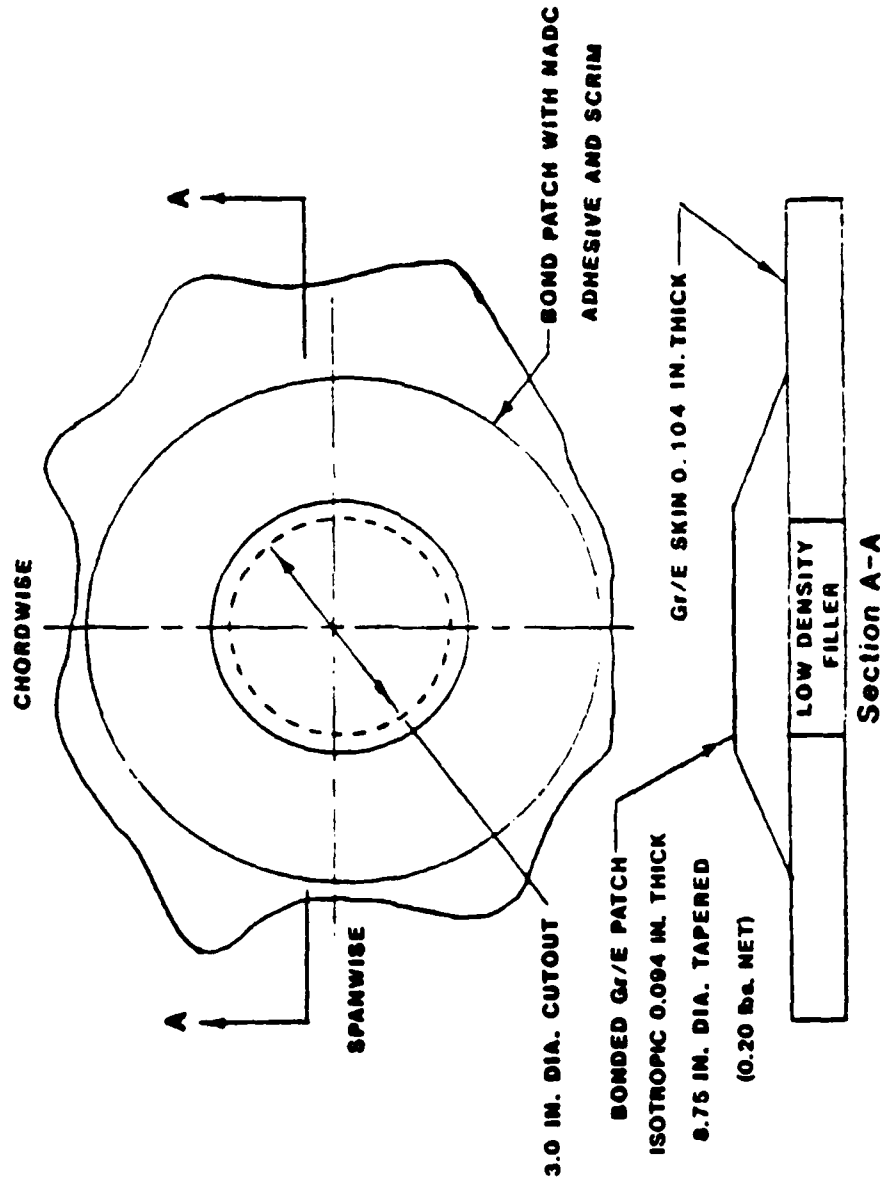


Figure 9. B-Staged External Bonded Repair

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